IMPROVING BOND STRENGTH THROUGH ACID ETCHING OF DENTIN AND BONDING TO WET DENTIN SURFACES

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ABSTRACT

A bonding system using moisture on the tooth surface can be an enormous benefit as obtaining dentin dryness in the mouth is nearly impossible. The author describes a system that bonds to both wet and dry surfaces.

Restorative dentistry needs a dentin-enamel bonding system that can:

- provide gapfree composite restorations;
- achieve rapidly developing high-bond strengths;
- be biocompatible;
- function in the presence of moisture;
- treat dentin and enamel simultaneously;
- treat multiple types of intraoral surfaces.

Producing a gapfree restoration is critical, especially in composite resin restorations. Brannstrom has shown that bacteria can rapidly colonize the gaps between composite resin restorations and the tooth interface.[1] Bergenholtz and others found that bacteria are directly correlated to pulpal pathosis.[2]

Bonding systems need to generate high-bond strengths in a short time. Typically, 24-hour bond strengths are the reported values, but no one waits 24 hours to begin inserting composite resins. Davidson and others showed that the polymerization shrinkage of composite resins places stress on bonding systems.[3] To resist the contraction force of polymerizing composites, bond strengths must develop rapidly. The need for biocompatibility is obvious, but it's likely that all current bonding systems are biocompatible.

Since dentin is inherently a wet substrate, the bonding system must function in the presence of moisture. The need for a common treatment of dentin and enamel is again obvious to the practicing dentist, who well knows there really is no other way to treat. Often it's very difficult to recognize where enamel ends and dentin begins. Keeping an enamel etchant off dentin all the time is not practical. Remember this when considering a bonding system that is detrimentally affected by the enamel etchant contacting the dentin surface.[4]

Finally it would be most convenient to the practitioner if one bonding system could be used to bond all types of surfaces. Rather than purchase one kit to bond enamel, another for metal and yet another for porcelain, it would be highly desirable to be able to use the the same bonding system for all surfaces with only small alterations. For this reason we should consider not dentin enamel bonding systems but rather adhesive systems.

Perhaps the most intriguing need is function on moist or wet dentin surfaces. Freshly cut dentin presents a difficult restorative medium for adhesive resin systems. The dentin substrate is inherently moist because of the tubular fluid. Reports have demonstrated that moisture typically deters the ability of resin bonding systems to adhere to dentin. Terkla and others[5] showed that two dentin bonding resin systems tested could not seal moist dentin.

Mitchem and others[6] noted that the bond strengths of Scotchbond (3M Dental Products), Tenure (Den-Mat) and Gluma (Columbus Dental) fell when tested under simulated physiological conditions. Andreaus and others[7] reported that water in dentinal tubules interfered with the adhesion of dentin adhesives. Tao and Pashley[8] reported that the bond

Figure 1. Maxillary left central incisor before restoration of dentin.

Figure 2. Cord is packed in gingival sulcus to prevent fluid contamination.

strength of Gluma and Scotchbond fell significantly when smear layers were removed and pulpal pressure was applied to a dentin surface. Prati and others[9] found that storage in simulated pulpal pressure caused a significant reduction in the bond strength of Clearfil Photobond (J. Morita) and Scotchbond 2 in deep dentin. Mitchem and Gronas[10] and Glasspoole and others[11] reported that the bond strength of Scotchbond 2 was diminished in the presence of dentinal moisture.

This report presents data demonstrating the improvement in bond strengths obtained with a particular bonding system when dentin is acid-etched and the primers are applied to wet dentin surfaces. The clinical technique is also illustrated.

MATERIALS AND METHODS

Sixty extracted human molars were selected for this study. They were free of obvious defects and used within two weeks of harvest. They were cleaned with a soap containing chlorhexidine and placed in a 3 percent glutaraldehyde solution for 24 hours. They were then rinsed and stored in tap water at 4 C until time of usage. For the experiment the teeth were embedded in an autopolymerizing acrylic resin in a stainless ring with an inside diameter of one inch. The teeth were ground on a model trimmer to expose adequate facial dentin surface for bonding.

The samples were stored in water at 37 C for 24 hours. After storage, the exposed dentin surfaces were wet-sanded with 320 grit sandpaper just before treatment. The teeth were randomly divided into three equal groups, each of which was divided into two subgroups.

The dentin-enamel bonding system examined was the All Bond 2 system (Bisco Dental). The system has available two concentrations of phosphoric acid semigel etchant conditioners: a 10 percent and a 32 percent concentration. The system also contains a Primer A and Primer B and a dual-cure capability unfilled bonding resin. Primer A is made up of NTG-GMA(N-tolyl glycine

TABLE 1

LIST OF MATERIALS

All-Etch (10% phosphoric acid semigel etchant)
Uni-Etch (32% phosphoric acid semigel etchant)
Primer A (batch 129161)
Primer B (batch 129171)
Dentin-enamel bond (batch 129091)

glycidylmethacrylate)/acetone and Primer B is BPDM (biphenyl dimethacrylate)/ acetone (Table 1).

The teeth were divided into three equal groups and were treated in the following manner:

■ Group 1. The smear layer was not removed. Dentin surfaces in 10 teeth were air-dried for 3 seconds with the tip of the air syringe about 3 centimeters from the tooth surface. A drop each of Primer A and Primer B were mixed together in a well. The primer mixture was applied to the dentin surface in a minimum of five consecutive coats. The surfaces were not dried between coats.

Each application was able to spread out on the surface before the next was made. After the fifth application, the primed surface remained undisturbed for 5 seconds and then was air-dried for 5 seconds. A layer of the dentin-enamel unfilled bonding resin (Bisco) was applied to the primed surface, air-thinned for 1 to 2 seconds and light-polymerized for 20 seconds with a Demetron 401 light-curing unit (Demetron Research). A Teflon mold 2.5 millimeters thick with a cylindrical matrix 4 mm in diameter was then clamped to the embedded tooth so that the matrix was over the treated dentin. The matrix was filled with a single increment of All-purpose composite (universal shade, Bisco Dental), and the composite was light-activated for 40 seconds.

On the second 10 teeth, the smear layer was rinsed but left visibly wet, or moist. The moisture presence was obvious, in that the dentin surface was shiny with moisture. A facial tissue was sprayed with air-water until it was wet and wiped the surface of the samples, removing only the excess water. (For a patient, simply use a disposable brush to rewet the dentin surface should it become dry.) A drop each of Primer A and B were mixed together in a well and applied to the wet dentin surface in a minimum of five consecutive coats as described. The primed surface was dried and the dentin-enamel bond applied. Composite cylinders were bonded to the treated dentin surface.

■ Group 2. The dentin was treated with 10 percent phosphoric acid gel etchant (All-Etch, Bisco Dental Products) for 30 seconds and rinsed for 5 seconds with an air-water mixture. On 10 teeth, the dentin was dried for 3 seconds with an air syringe at full blast at a distance of about 2 cm. On the remaining 10 teeth, the

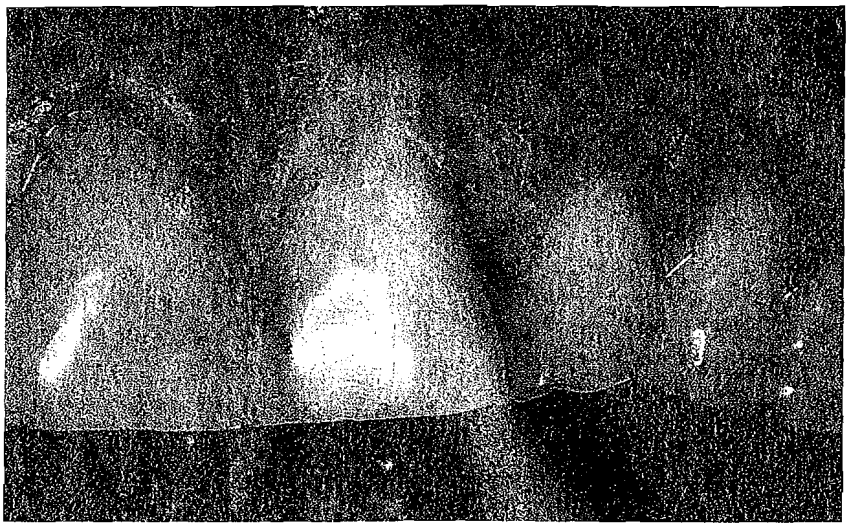


Figure 3. Cavity is prepared with light bur treatment to enhance retention.

Figure 4. Preparation is treated with semigel etchant and rinsed with air-water.

Figure 5. Matte appearance of surface shows area too dry to be treated.

dentin was left wet as described in group 1. Primer A and B and the dentin-enamel bonding resin were applied as described. It sometimes took more than five coats of the Primer A and B mixture to produce a glossy coating of dentin. We placed enough applications of the A and B mixture to achieve this appearance. Composite cylinders were bonded to the dentin surfaces as previously described.

■ Group 3. The dentin in this group was treated with 32 percent phosphoric acid gel etchant (Uni-Etch, Bisco Dental) for 20 seconds and rinsed for 5 seconds with an air-water mixture. On 10 teeth, the dentin was dried for 3 seconds with an air syringe as in group 2. On the remaining 10, we wiped the dentin with a wet tissue to remove only the excess water as in group 1. Primer A and B and the dentin-enamel unfilled bonding resin were applied to the dentin as described in group 2. Composite cylinders were bonded to the samples as above.

For a control group, 10 incisors were embedded in acrylic and ground to produce adequate enamel surface for bonding. The enamel was treated with the 32 percent etchant for 20 seconds, rinsed and dried. Primer A and B and the bonding resin were applied as in group 3. Composite cylinders were applied as previously described.

The matrices were removed from the teeth and the samples were stored for 24 hours in water at 37 C. Shear bond strength was measured on an Instron machine (Model no. 1123) with a crosshead speed of 5 mm/minute and recorded. We used a sharp blade to shear the samples with the blade parallel and immediately adjacent to the bonded dentin surface.

RESULTS

The best results were obtained when the dentin was acid-etched and the primer mixture applied to wet dentin surfaces (Table 2). The lowest bond strengths occurred when the primer mixture was applied to a dry smear layer. In all cases, we obtained higher bond strengths when the primer mixture was applied to a wet dentin surface. Failure modes were examined with a light microscope.

In the 10 percent etch/wet and 32 percent etch/wet groups, all samples failed cohesively within the body of the dentin. In the 10 percent etch/dry and 32 percent etch/dry groups, four of 10 samples failed cohesively in the dentin. All samples bonded to a smear layer experienced adhesive failure between the resin and the smear layer. Two-way ANOVA analysis of each of the

TABLE 2

BOND STRENGTHS WITH VARIOUS SURFACE TREATMENTS

	SURFACE			
	DRY		WET	
GROUP	Mpa (S.D.)	CV	Mpa (S.D.)	CV
320 grit smear layer	11.7 (4.8)	46.3%	24.1 (2.1)	8.8%
10% H3PO4 30s	24.3 (5.2)	21.4%	34.3 (2.4)	7.0%
32% H3PO4 20s	20.7 (10.8)	52.2%	36.5 (1.6)	4.4%
32% H3PO4 20s (enamel)	31.9 (4.8)	15.0%		

All groups N = 10.
CV = coefficient of variance.

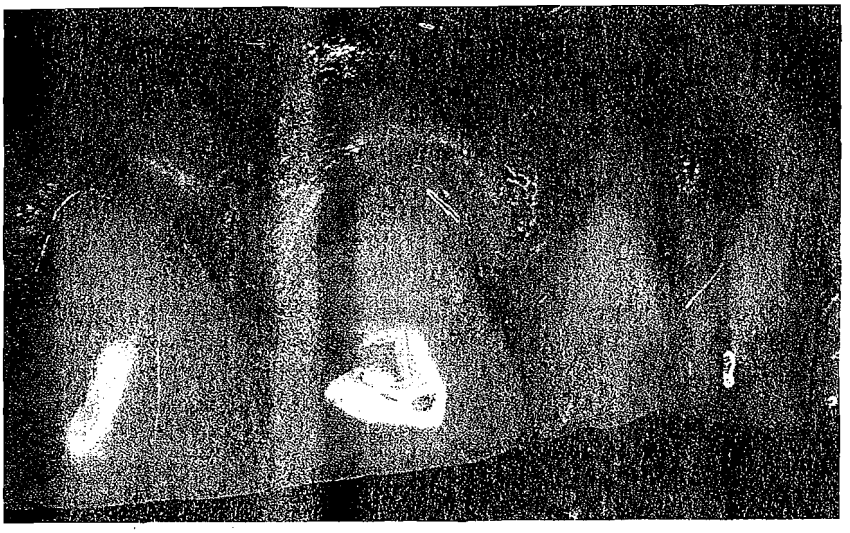


Figure 6. Preparation surface must be wet for higher bond strength.

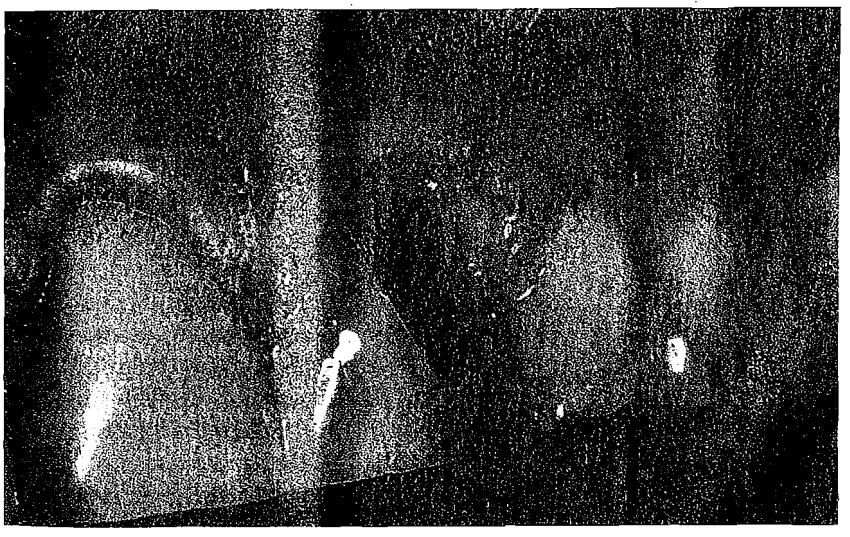


Figure 7. Primers are applied directly on wet dentin.

groups (etch vs. no etch, wet vs. dry) shows that in each case the bond strength of materials was significantly higher to the wet than the dry dentin surface ($P<.05$). The coefficient of variation, however, was lower for all of the wet-bonded samples. This may indicate a greater reliability of the bond.

DISCUSSION

The natural state of dentin in vital teeth includes fluid in the tubules. This tends to create a moist surface on dentin. Nordenvall reported that it is difficult to dry vital dentin and to maintain a dry state because of the outward flow of dentinal fluid.[12] A dentin-enamel bonding system that can adhere to a wet surface has a great advantage. It's difficult to determine that the tooth surface is completely dry before applying conventional dentin bonding materials that demand dryness. This is particularly true in posterior areas and any areas to which access is compromised or difficult.

The most intriguing aspect of the material being studied is that we obtained the best adhesion values with wet surfaces. As we explained in an earlier report, mixing the acetone in the primer mixture with the water on the dentin surface caused the vapor pressure of the water to rise and created some evaporation.[13] Mixing the acetone/resin mixture with the water also reduces the surface tension of the water so that the mixture seems to "chase" the water.

This results in the primer layer being laid down in intimate contact with the dentin surface; that is, the intertubular dentin, peritubular dentin and presumably the dentinal tubule lumina are adapted to more thoroughly when the primer mixture is applied to the wet surface. This system can generate gapfree restorations in vitro and in vivo, and the resin penetration in vivo was the same as in the in vitro state.[14] (In this report, wetness or moisture refers only to dentinal fluid, water or a disinfectant solution. Salivary contamination is unacceptable as saliva contains viscous proteinaceous material as well as bacteria.)

It has also been reported that the first version of the All Bond system can bond to wet enamel as well as to wet dentin.[15] Bond strengths obtained in that report were on the order of 29 to 32 Mpa. The current data show this system's ability to generate bond strengths to dentin in excess of those to enamel. (Data to be published show this to be the same with the All Bond 2 system.)

All discussions of dentin bonding materials should contain some reference to an

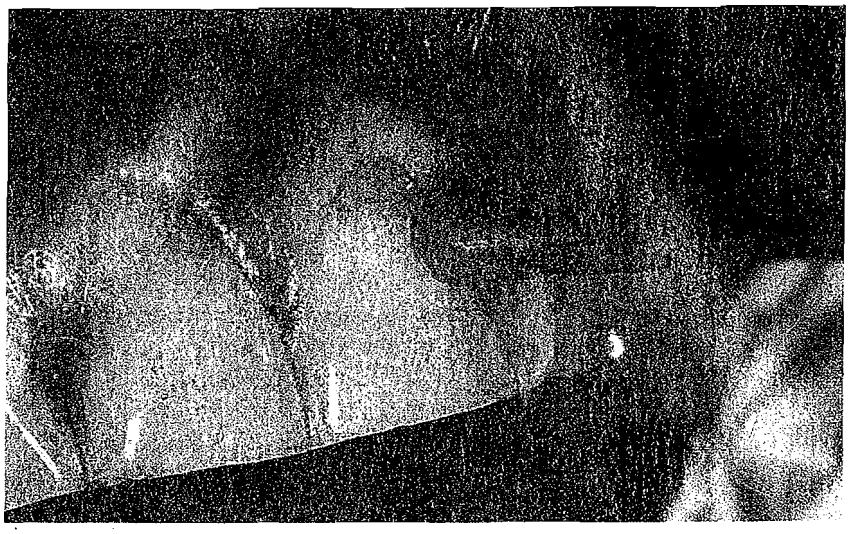


Figure 8. Five coats of primer are applied without drying. Preparation is gently air-dried.

Figure 9. The dentin-enamel bonding resin is applied to primed surface.

etched enamel standard. The simple disclosure of dentin bond strength values without that standard can be difficult to interpret. This bonding system uses a phosphoric acid dentin-enamel conditioner. Typical reports on dentin bonding cite two references that noted that applying phosphoric acid to dentinal surfaces reduced bond strengths.[16,17] The authors used a hydrophobic unfilled BIS-GMA autopolymerizing resin on etched dentin surfaces with 50 percent phosphoric acid on the dentin for up to 2 minutes.

Both the concentration of acid and time of application have changed considerably. It has still been reported, however, that the bond strengths of even current bonding systems may be diminished by using phosphoric acid as the dentin conditioner.[4] The greatest controversy in this system stems from the belief that applying phosphoric acid to cut dentin will harm the pulp. This was concluded in some early studies examining the alleged effects of acid on dentinal surfaces.[18,19]

In those studies, Class V cavity preparations were cut into teeth and the cavities in the control group were restored with zinc oxide-eugenol. In the experimental group, the cavities were acid-etched with 50 percent phosphoric acid for 1 minute and then rinsed, dried and filled with ZOE. The pulps in the experimental groups showed a negative reaction to something, and the authors concluded that that something was the acid treatment.

But a review of the literature showed that the control material (ZOE) is cytotoxic when placed against living tissue.[20-27] In addition, acid treatment of dentin increases its permeability.[28] Eugenol can diffuse through intact dentin,[29] and Brannstrom and coworkers have shown that ZOE-type materials have damaging effects on the pulp when placed on thin remaining dentin in vivo.[29-32] In an earlier report,[33] I speculated that the acid treatment of the dentin alone did not cause the pulpal reactions seen in those studies. It was more likely that ZOE application to the etched permeable dentin caused the reactions as eugenol (a phenol) diffused into the pulp more readily than when a smear layer was present, as in the unetched group. Considering these findings, we see that the major concern is not the acid treatment of dentin but the material placed on the dentin after etching and its ability to seal the dentin.

Recent reports support this contention. At the most recent AADR meeting, a paper showed

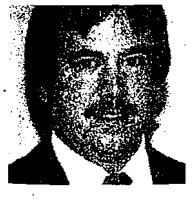
the All Bond bonding system as biocompatible when applied after 37 percent phosphoric acid treatment of freshly cut dentin for up to 2 minutes. The pulp reaction to this treatment was less than that to the ZOE unetched controls.[34] Another presentation noted the same results.[35] Dentin was treated with 37 percent phosphoric acid and the teeth were restored with New Bond (J. Morita) and a light-cured composite. These findings can significantly affect the future of adhesive restorative dentistry.

TECHNIQUE

We restored a maxillary left central incisor (Figure 1). There was tooth tissue absent from the cervical areas of the tooth, and the dentinal surfaces were shiny. These shiny dentin surfaces pose a difficult restorative problem as it's harder to restore than normal dentin.[36] We recommend preparing sclerotic dentin lightly with a diamond bur to enhance retention and using a bonding system to produce gapfree composite resin restorations in vivo.[14]

Steps for restoring this maxillary left central incisor follow:

- Pack cord in the gingival sulcus to prevent fluids from contaminating the field (Figure 2).
- Prepare the cavity conservatively by light treatment with a diamond bur (Figure 3). No mechanical retention is used.
- Treat the preparation with the 10 percent semigel etchant for 30 seconds on both dentin and enamel and rinse thoroughly with air-water (Figure 4).
- If desired, use the 32 percent gel etchant on the dentin and enamel for 20 seconds in lieu of the 10 percent etchant. Figures 5 and 6 show the contrast between wet and dry prepared surfaces. As this bonding system obtains higher bond strengths to wet dentin, be sure the preparation surface appears wet as shown in Figure 6. If the preparation surface becomes too dry, it will have a matte appearance (Figure 5). If this happens, remoisten the surface of the preparation with water or an antimicrobial solution (for example, Tubulicid Red, Global Dental Products) with a disposable brush.
- Mix together a drop of Primer A and Primer B in the well and place directly on the wet dentin (Figure 7).
- Apply five consecutive coats



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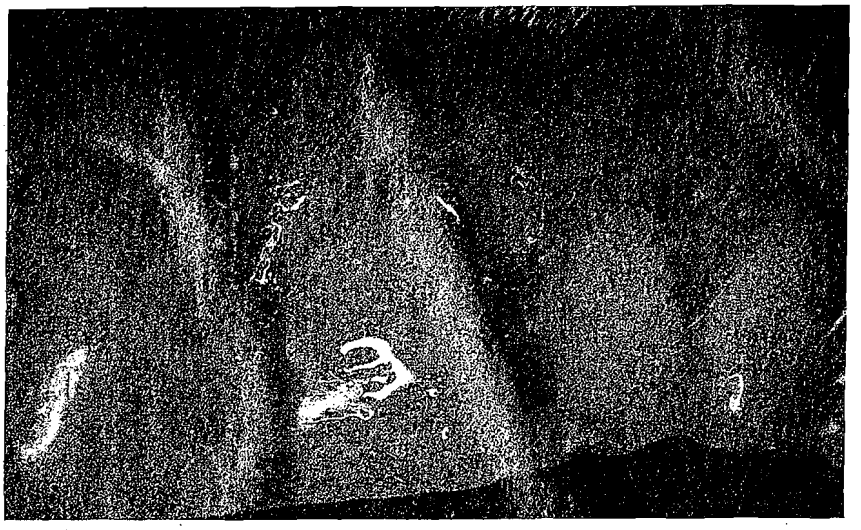

Figure 10. Appearance after resin is applied and air-thinned.

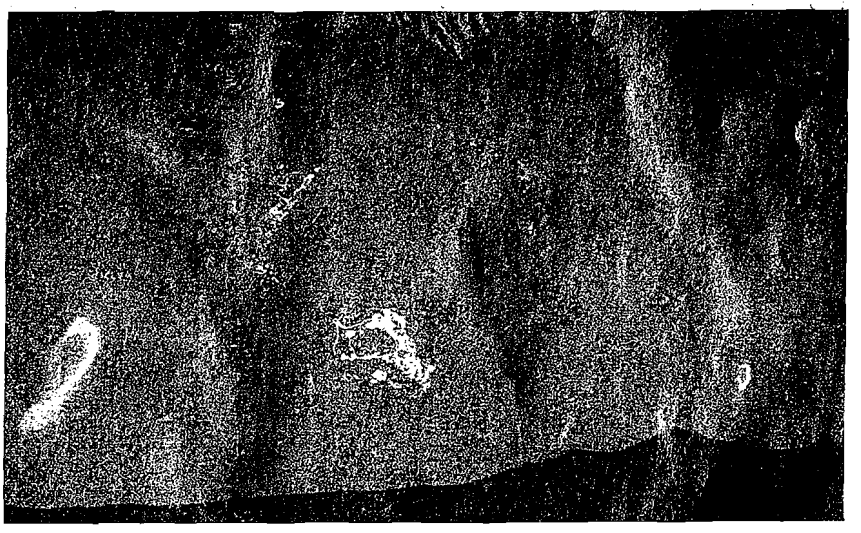

Figure 11. Composite resin is applied incrementally and light-polymerized.

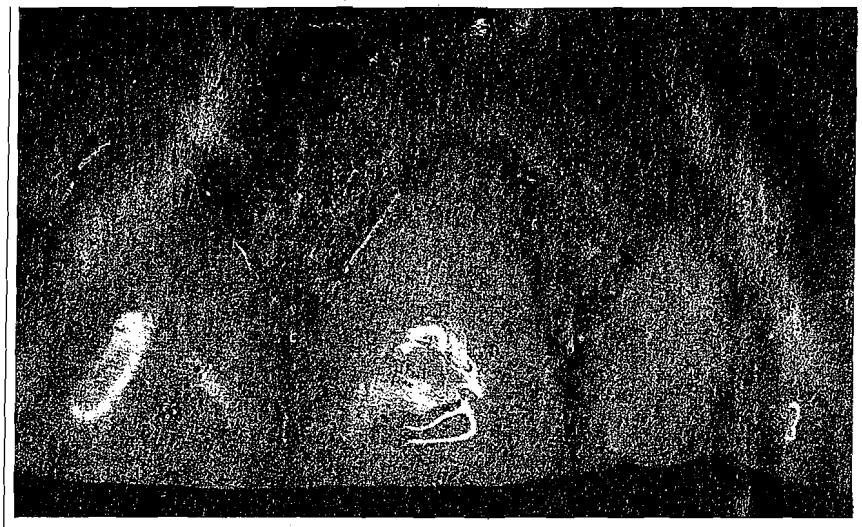

Figure 12. Completed restoration at one-year recall.

(or more—use up the mixture) without drying between coats. After a few seconds, gently air-dry the surface for 2 to 3 seconds to remove any remaining volatiles (Figure 8).

- Apply the dentin-enamel bonding resin to the primed surface and air-thin (Figure 9,10). Light-polymerize for 20 seconds.
- Place composite resin (Herculite XR-V, Kerr Dental) incrementally and light-polymerize appropriately (Figure 11). Finish the filled preparation with burs and disks.

The completed restoration is shown in Figure 12. Adjacent to the newly completed restoration is a similarly restored tooth at one-year recall. I've used this system successfully in the past three years, retaining 58 of 58 Class V restorations over a 30-month period with no post-operative sensitivity. The All Bond system has also been shown to bond to all typical intraoral substrates.[37,38]

CONCLUSION

This report shows that a dentin-enamel bonding system can not only bond to wet dentin surfaces, but also offer the best results when the dentin surface is wet. In addition, the best shear bond strength data are obtained through acid-etching the dentin. This provides a significant reduction in technique sensitivity. The clinician may apply the gel etchant to both the dentin and enamel at the same time—this is far easier than trying to etch the enamel alone. After rinsing the etchant from the tooth surface, remove only the excess water with a short blast of air—desiccation is not required.

High in vitro bond strengths result from this type of treatment, and the bonding system is biocompatible. It must be made clear that this report does not indicate endorsement of dentin treatment with any phosphoric acid and the indiscriminate placement of any bonding system. It has been reported that phosphoric acid treatment can diminish the bond strength of many bonding systems,[4] and the manufacturers' instructions must be followed for each system. However, there are obvious advantages to using a system that can treat dentin and enamel at the same time with phosphoric acid while improving the bond strength to both the enamel and the dentin. ■

Information about the products mentioned in this article may be available from the author. Neither the author nor the American Dental Association has any commercial interests in the products mentioned in this report.

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